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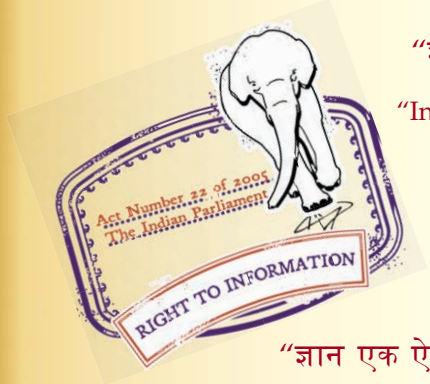
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IS 11177 (1985): 4-nitro-2-toluidine [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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SPECIFICATION FOR 4-NITRO-2-TOLUIDINE

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SPECIFICATION FOR 4-NITRO-2-TOLUIDINE

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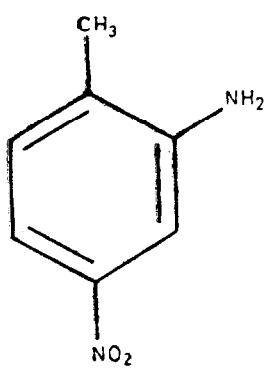
Indian Standard

SPECIFICATION FOR 4-NITRO-2-TOLUIDINE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 January 1985, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 4-Nitro-2-toluidine ($C_7H_8O_2N_2$) is an important dye intermediate. It is prepared by nitration of 2-toluidine sulphate and then hydrolysis. It is also commercially known as Fast Scarlet G Base. It is represented by the following structural formula:



4-NITRO-2-TOLUIDINE

(Moleclular Mass 152)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical value (*revised*).

1. SCOPE

1.1 This standard prescribes requirements and the methods of sampling and test for 4-nitro-2-toluidine.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of yellow-coloured powder or paste free from visible impurities.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 4-NITRO-2-TOLUIDINE

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in Appendix A
(1)	(2)	(3)	(4)
i)	Assay, percent by mass, *Min*	98	A-1
ii)	Melting point, °C	102 to 103	A-2
iii)	Moisture, percent by mass, *Max*	1·5	A-3
iv)	Impurities (5-nitro-2-toluidine), percent by mass, *Max*	0·5	A-4

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1979*) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer and his recognized trade-mark, if any;
c) Tare, net and gross mass; and
d) Batch number.

*Specification for steel drums (galvanized and ungalvanized) (*second revision*).

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for the determination of 4-nitro-2-toluidine shall be conducted on each of the individual samples.

4.2.2 Tests for the determination of remaining characteristics, namely, description, melting point, and moisture shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of 4-nitro-2-toluidine if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics (*see* **4.2.2**) tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirements given in **2.1** and Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in Appendix A, as indicated in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in test.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

(*Table* 1, and *Clause* 5.1)

METHODS OF 4-NITRO-2-TOLUIDINE

A-1. ASSAY (BY NITRITE VALUE)

A-1.1 Reagents

A-1.1.1 *Hydrochloric Acid* — *See* IS : 265-1976*.

A-1.1.2 *Potassium Bromide* — analytical reagent grade.

A-1.1.3 *Sodium Nitrite Solution* — 0·2 N.

A.1.1.4 *Starch Iodide Papers*

A-1.2 Procedure

A-1.2.1 Weigh accurately 0·7 to 0·8 g of sample in l-litre beaker. Add 20 ml of concentrated hydrochloric acid and warm the solution, if necessary to effect complete dissolution. Cool to room temperature. Add 400 ml of distilled water and little quantity of ice to cool the solution 0 to 5°C. Add 5 g of potassium bromide and titrate against standard sodium nitrite. When a drop of mixture from the beaker is placed on starch iodide paper, instantaneously a blue ring is developed. Confirm the end point after 1 minute, without addition of sodium nitrite solution. Note the volume of standard sodium nitrite solution consumed.

A-1.3 Calculation

$$\text{Assay (on dry basis), percent by mass} = \frac{V \times N \times 0{\cdot}152}{M} \times 100$$

where

V = volume in ml of standard sodium nitrite solution consumed,

N = normality of standard sodium nitrite solution, and

M = mass in g of the sample taken for the test.

A-2. DETERMINATION OF MELTING POINT

A-2.1 Determine the melting point of the material as prescribed in **8** of IS : 5299-1969†.

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*Specifiation for hydrochloric acid (*second revision*).

†Methods of sampling and tests for dye intermediates.

A-3. DETERMINATION OF MOISTURE

A-3.1 Determine the moisture content of the material as prescribed in **9.3** of IS : 5299-1969*.

A-4. DETERMINATION OF IMPURITIES

A-4.0 Outline of the Method — The probable impurity is 5-nitro-2-toluidine and it is determined by thin layer chromatography.

A-4.1 Apparatus

A-4.1.1 *Chromatographic Chamber* — 22 × 10 × 22 cm.

A-4.1.2 *Micropipette* — of 10 μl-capacity.

A-4.1.3 *Thin Layer Chromatographic Glass Plates* — 20 × 20 cm.

A-4.1.4 *Adjustable Thin Plate Applicator*

A-4.1.5 *Chromatographic Spray Bottle*

A-4.1.6 *Oven* — with a thermo regulator.

A-4.1.7 *Weighing Bottle*

A-4.2 Reagents

A-4.2.1 *Solution of 5-Nitro-2-Toluidine in Methanol* — 0·02 percent and 0·4 percent (*m/v*) (reference solution).

A-4.2.2 *Eluent* — Benzene : Methanol (98 : 2).

A-4.2.3 *Coupling Reagent* — 0·5 percent methanolic solution of American Base (N-1-Naphthylethylenediamine dihydrochloride).

A-4.3 Procedure

A-4.3.1 *Preparation of the Chromatographic Plates* — Mix 50 g of neutral silica gel with 85 to 90 ml of water for three minutes. Then, by means of the applicator, coat the plate with the silica gel layer of thickness not exceeding 0·25 mm and place it in the oven maintained at 110°C for 90 minutes. By means of a needle, mark a thin line width wise on all plates at about 3 cm from the upper edge. Also remove a band of about 5 mm width silica gel from either of the lateral sides of each plate.

*Methods for sampling and test for dye intermediates.

A-4.3.2 *Preparation of the Sample Solution* — Homogenize the sample for a few minutes and weigh by means of a weighing bottle 4·0 g of the material and transfer to a 100 ml volumetric flask. Dissolve in methanol and make up the solution to the mark by adding additional quantity of methanol.

A-4.3.3 Spot 10 μl of the sample solution (**A-4.3.2**) and those of reference solutions of 5-nitro-2-toluidine (**A-4.2.1**) on the thin layer plate. Place the developer in the chamber. Close the chamber with its lid and allow to achieve equilibrium. Now place the plate carefully in the chamber and allow the eluent to run in ascending manner for about 25 to 30 minutes. Remove the plate from the chamber and dry off the solvent in an air drier. Hold the plate for 20 seconds in nitrous acid fumes (formed by adding about 5 g of sodium nitrite to 50 ml of 5 N hydrochloric acid in a 5-litre beaker. Allow the plate to stand for 1 minute to remove excess nitrous fumes. Spray with 0·5 percent of methanolic solution of American base (**A-4.2.3**).

A-4.3.3.1 By visual comparison, the spot corresponding to 5-nitro-2-toluidine in the sample shall have equal or less intensity than that of reference sample of 0·5 percent 5-nitro-2-toluidine and there shall be no other impurities on the thin layer glass plate.